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THE SPECIFIC HEAT OF THERMOSETTING POLYMERS (U)

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THE SPECIFIC HEAT OF THERMOSETTING POLYMERS

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ABSTRACT: Specific heat, as a function of temperature has been determined over the range of 30°C to 160°C for a number of thermosetting polymers. The data were obtained by means of an adiabatic calorimeter. In agreement with theory, the specific heats of the polymers showed a progressive increase with increasing temperature. In many cases large increases in specific heat have been observed which are indicative of second order transitions within the polymer.

CHEMISTRY RESEARCH DEPARTMENT
U. S. NAVAL ORINANCE LABORATORY
White Oak, Maryland

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This report contains data on specific heats as a function of temperature for a number of thermosetting polymers. In many cases the results are indicative of second order transitions which occur within the polymer. This study was made under Project N10a-1-56 as part of a general polymer investigation.

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By direction

ACKNOWLEDGEMENTS

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THE SPECIFIC HEAT OF THERMOSETTING POLYMERS

INTRODUCTION

1. A knowledge of the specific heat of thermosetting polymers is becoming more important with increasing use of these materials, often under high temperature conditions. Values of specific heat (hereafter referred to as C_p) determined as a function of temperature can be used in calculating heats of polymerization of polymers using the techniques of differential thermal analysis (Reference 1) and adiabatic calorimetry (Reference 2). In addition, thermal diffusivities may be calculated from the C_p data when the thermal conductivity and the density are known. The changes of C_p with temperature are indicative of changes in the electrical and mechanical properties of these materials and are frequently related to the second order transitions.

2. Some work has been done on determining the C_p of synthetic polymers but a systematic study of the thermal properties of thermosetting polymers has not been previously undertaken. Dole and co-workers (Reference 3) have determined C_p for polyethylene and a number of other synthetic polymers, and polystyrene has been studied by Ueberreiter and Otto-Laupenmuhlen (Reference 4). Gast (Reference 5) has studied the C_p of polyvinyl chloride. Furukawa, Bekkedahl and co-workers at the National Bureau of Standards have determined C_p for a number of polymers (References 6 and 7). In addition, a number of values of C_p have been reported but with no information as to the temperature at which the determination was made or of the method that was used.

3. A calorimeter was constructed for the purpose of determining the C_p of thermosetting polymers. The design of this instrument was based on the maintenance of adiabaticity between the polymer sample in the calorimeter vessel and the calorimeter wall during the C_p determination. The temperature of the cured polymer sample was increased continuously by means of an embedded resistance coil which was connected to a source of energy. Adiabatic conditions were maintained by electrically heating the calorimeter wall at such a rate that the wall temperature was within 1°C of the temperature of the polymer sample at all times during the determination. This calorimeter was designed to obtain engineering data with a minimum of effort and could be operated by a single technician. The data obtained were estimated to be accurate to within 5%.

4. The work reported upon herein was part of a long-term study of thermosetting polymers being conducted in an effort to obtain a more basic understanding of the thermal, electrical and mechanical properties of these materials. In addition, a study was being conducted using thermal and electrical methods to get information on the polymerization characteristics of these compounds. The extent to which the physical properties of the solid polymer are influenced by the manner in which the polymerization is conducted was also being studied. The results of this C_p

investigation indicate that C_p determination made as a function of temperature are real functions of the structure of the polymer and can be used in studying the thermal and mechanical properties of thermosetting polymers.

THEORY

5. The theory of the specific heat of linear polymers has received a limited treatment by Boyer and Spencer (Reference 7). These investigators attributed the increase in C_p with temperature to increasing degrees of freedom within the polymer. The molecules in the solid vibrate about their equilibrium positions with an amplitude which increases with increasing temperature. As the temperature is raised these new degrees of freedom are slowly activated and absorb heat energy. The observed effect of these new degrees of freedom, is the increase in C_p of the polymer. A theoretical treatment of the C_p of thermosetting polymers has not been made. In this class of polymer the problem is even more complex as the entire polymer is crosslinked and vibrations and rotations occur only with difficulty.

6. The increase in C_p with temperature is only of limited interest when compared to the fact that in many polymers there are rather abrupt increases in C_p over relatively narrow temperature ranges. This rapid increase in C_p has been observed by many investigators (References 3, 7 and 8) and can be attributed to a second order transition, T_g , occurring within the polymer. This transition which occurs upon heating a polymer manifests itself as a change in the temperature dependence of a large number of physical properties. At this temperature point, or more correctly, in this temperature range, a discontinuity occurs in the thermodynamic quantities such as specific heat, thermal conductivity and thermal expansion. As the temperature is raised through this region, a process takes place not involving latent heat, which results in larger values of the thermodynamic quantities and in a change in the general physical properties of the material. In general, when the material is above the transition temperature it becomes softer and more flexible as contrasted to its hard brittle character at lower temperature.

7. There is no simple physical explanation that will completely describe T_g in terms of the structure of a polymer and which can be applied to all polymers. For linear polymers, T_g increases approximately in proportion to the secondary valence force per unit length of polymer chain (Reference 7). However, bulky groups and strong polar groups lead to higher T_g values (Reference 7). It has been considered by some workers in this field that T_g is an internal melting point, above which the polymer still preserves the external characteristics of a solid while behaving in part like a liquid. As the polymer is heated and T_g is reached, sections of long polymer chains move further apart and are able to move more freely about the length of the chain (Reference 7). This new vibration and

rotation which occurs at T_g can account for the increase in C_p . Alford and Dole (Reference 9), who studied T_g in polyvinyl chloride, concluded that the large increase in C_p at T_g could not be entirely the result of additional vibrations in the solid; other factors such as interchain interactions involving a potential energy were suggested as being involved.

8. Van Amerongen (Reference 10) has concluded that the temperature range of T_g in linear polymers is mainly determined by the strength of the secondary valence energies and by the flexibility of the chain. In thermosetting polymers the higher values of T_g are primarily determined by the extent of crosslinking. This has been shown by Fox and Loshack (Reference 11) who pointed out that crosslinking involves the exchange of secondary bonds for primary bonds.

EXPERIMENTAL

A. Method

9. The adiabatic calorimeter, the calorimeter vessel, the temperature monitoring and controlling instrumentation, and a detailed description of the techniques used to determine the C_p of thermosetting polymers have been previously described (Reference 12). In addition, the calibration of the calorimeter was described and the results of a C_p determination on polyethylene were compared with the results obtained on the same material by Wunderlich and Dole (Reference 3).

10. The adiabatic calorimeter consists of three closed concentric cylinders separated by dead air spaces. Placed around the inner cylinder is a heating coil of nichrome resistance wire. The two other cylinders and the dead air spaces serve to insulate the inner cylinder and prevent loss or gain of heat during a C_p determination. The cylindrical polymer sample containing an encapsulated heating coil and thermocouples is placed in the inner cylinder of the calorimeter. The heating coil in the polymer sample is connected to a source of power and the temperature of the sample is thus raised at a uniform rate. This increase in temperature is measured at 300 second intervals by means of the encapsulated thermocouples. Adiabaticity is maintained by heating the inner cylinder of the calorimeter at a rate so that its temperature is always within 1°C of that of the polymer sample. This prevents loss or gain of heat by the sample.

11. In each case the polymer was extensively postcured before C_p was determined. Each C_p determination was conducted in duplicate and in some cases additional determinations were made. The C_p was calculated by means of Equation 1.

$$C_p = \frac{I^2 R t}{4.185 M (\Delta T)} \quad (1)$$

Where I is the current in amperes, R is the temperature dependent resistance of the heating coil in ohms, t the time in seconds, M the mass of the polymer in grams, (ΔT) the rise in temperature of the polymer in $^{\circ}\text{C}$, and the constant 4.185 is the mechanical equivalent of heat in joules per calorie.

B. Materials

12. The amorphous thermosetting polymers studied in this investigation consisted of a number of commercially available compounds. Many of these compounds were casting resins. All the polymers were prepared for study by the methods described in Reference 12. In addition to the thermosetting polymers, two thermoplastic polymers, polyethylene and polystyrene were used to check the accuracy of the calorimeter. The polymers studied, the polymer type, the curing agent used and the manufacturer are shown in Appendix I.

C. Results

13. Figures 1 to 6 are plots of the C_p as a function of the temperature for the polymers studied. Inspection of these plots shows that in each case the C_p increased with an increase in temperature. In one case (Epon 828 cured with *m*-phenylene diamine) this increase in C_p was approximately linear. This indicates that no sharp transitions occur within the polymer over the temperature range considered. However, in many of the polymers studied the C_p , after an initial linear interval, suddenly increases and then assumes an approximately constant value which is much less temperature dependent. In two cases (polyamide-EPL 2795 and diallyl phthalate) the C_p changes throughout the temperature range considered. The rate of change was not constant yet no second order transition was indicated.

14. Published values of T_g for thermosetting polymers are nonexistent, but in the case of polystyrene, a thermoplastic polymer, Boyer and Spencer (Reference 7) reported a T_g value of 81°C . The value of $81-82^{\circ}\text{C}$ (Figure 6) is therefore in excellent agreement with the published value. The temperature range over which T_g occurred is shown in Table 1.

DISCUSSION

15. It has previously been noted that most polymers show an increase in C_p with temperature, and those polymers studied in this investigation all show this increase with temperature. This increase in C_p with temperature is however, of little significance when compared with the fact that in many of the thermosetting polymers studied there were rather abrupt increases in C_p over relatively narrow temperature ranges. These abrupt increases were usually followed by temperature ranges over which the increase in C_p was small and the rate approximately constant. These increases in C_p can be explained as due to second order transitions which

are occurring within the polymer (References 6 and 7). These transitions are shown in Table 1.

16. In an amorphous thermosetting polymer, T_g will usually take place over a temperature range. An example of this is shown in Figure 5 when T_g for Epocast R-155 occurs over a 28°C range. It is possible that in some polymers limited transitions can occur at lower temperatures due to the acquisition of limited mobility by portions of the main chain or by side groups (Reference 13). However, T_g is catastrophic and unambiguous, the other limited transitions occur at lower temperatures and the magnitude of such transitions when compared to T_g on C_p versus temperature plots will be very small (Reference 13).

17. The magnitude and the sharpness of the change of C_p at T_g in thermosetting resins are interesting. For example, in Figure 5 Epocast R-155 shows an increase in C_p from $0.40 \text{ cal/gm}^\circ\text{C}$ at 81°C to $0.58 \text{ cal/gm}^\circ\text{C}$ at 105°C . The other extreme is shown by Paraplex P-43 polyester resin shown in Figure 1. This resin shows an increase in C_p at T_g , but the increase is very small and sharp. At 127°C C_p is $0.47 \text{ cal/gm}^\circ\text{C}$ and increases to a value of $0.49 \text{ cal/gm}^\circ\text{C}$ at 130°C .

18. The observed T_g points for epoxide polymer systems can be correlated with heat distortion temperatures, D_h . The exact relationship between T_g and D_h is obscure, but usually D_h occurs at a slightly higher temperature than T_g . It appears likely that the two points are closely related, T_g being characteristic of the micro properties of a material and being a more fundamental measurement, while D_h is characteristic of the macro properties. Horner, Cohen and Kohn (Reference 14) have determined D_h for a number of epoxide polymer-catalyst systems. In the case of Epon 828 and *m*-phenylene diamine, D_h was found to be in the range of 150 - 159°C for an extensively postcured polymer. No T_g was found up to 160°C which is the limit of the calorimeter. In the case of Epon 828 with tris(dimethylaminomethyl)phenol tri(2-ethyl hexoate), D_h was found to be at 93°C . The results of this study indicate that T_g was complete at 70°C . In the two systems compared above, neither the curing cycles nor the amounts of curing agent used by Horner and co-workers were exactly the same as for the polymers used in the C_p determinations so that an exact comparison cannot be made. Weiss (Reference 15) has found D_h for Epon 828 cured with 56.5% of dodecenyl succinic anhydride to be 78°C . This is in agreement with the results presented here where T_g occurred at about 60°C . The effect of plasticizers is to lower the temperature at which T_g and D_h occur (Reference 10).

19. Those polymers that do not show indications of a second transition over the temperature range studied are of considerable interest. The fact that a transition is not observed suggests that T_g occurs at a temperature either below 25°C or above the maximum temperature attained by the calorimeter during the C_p determination. The latter case is probable with Epon 828 cured with *m*-phenylene diamine. With polyethylene, diallyl phthalate and polyamide-EPL 2795 the problem is more complex. It appears that in these polymers large numbers of structural groups do not suddenly become

activated as the temperature is raised. These three polymers have D_h of 75°C (Reference 16), 121°C (Reference 17) and 60°C, respectively (Reference 18). It is suggested that polymers in which T_g is not observed by C_p measurements, and in which it can be established that T_g does not occur below 25°C or above 160°C, can be considered to have a broad T_g which occurs over a very wide temperature range and is of relatively small magnitude.

20. Two polymers containing an SiO_2 filler were studied. The amount of filler present was determined by chemical analysis. The C_p of duPont 820-001 which contains 15% of SiO_2 , is shown in Figure 5 and the C_p of Hysol 6020-105 which contains 31.8% of SiO_2 is shown in Figure 2. It appears that this calorimeter can be used to determine C_p as a function of temperature for any polymer containing a filler, the only requirements being that the percentage of filler be not too high so as to make the viscosity of the liquid unpolymerized polymer too great for satisfactory pouring into the calorimeter vessel.

21. In the case of polystyrene a previous determination of the C_p as a function of temperature had been made by Ueberreiter and Otto-Laupenmuhlen (Reference 4). Their results showed that in the region below T_g the C_p of polystyrene was not a function of the molecular weight. Samples having an average molecular weight of 860, 2300 and 3650 were studied and between -16°C and +14°C the results of three separate C_p determination on the three samples yielded results that were almost superimposable. However, upon increasing the temperature the sample having an average molecular weight of 860 showed a T_g starting at about 25°C while the samples having an average molecular weight of 2300 and 3650 began to show a T_g at 65°C and 75°C, respectively. This illustrates the dependence of T_g on the chain length of the polymer. After T_g was complete the C_p values increased at a constant rate, but the sample having the highest molecular weight showed somewhat higher values of C_p .

22. The data shown in Figure 6 for polystyrene in the range of 50° - 80°C are almost identical with those of Ueberreiter and Otto-Laupenmuhlen. This is an excellent check on the calorimeter used here. However, after T_g occurs at 81 - 82°C the results in Figure 6 show a progressive increase in C_p and the values were somewhat higher than those found in the previous study. No information was available as to the average molecular weight of the polystyrene used in this investigation. From the fact that T_g occurred at 81 - 82°C it can be estimated to be high ($> 10,000$). The previous work has shown that after T_g had been reached the C_p was somewhat dependent on the average molecular weight and since the results here for C_p were somewhat higher, it can be concluded that the average molecular weight was high. The polystyrene used in this investigation had a D_h of 85°C (Reference 19).

23. Epon 828 resin was cured with three different amine curing agents and a comparison of the results of the C_p determinations for

the three systems is of interest. In the first system (Figure 1) m-phenylene diamine was the curing agent, and as mentioned previously, this system showed a progressive increase in C_p from about 0.33 cal/gm°C at 40°C to about 0.51 cal/gm°C at 160°C. This increase is almost linear and no T_g was observed. In the second system (Figure 3) diethylamino-propylamine was the curing agent, and this system showed both an increase in C_p and a T_g occurring over a wide temperature range, terminating at about 115°C. Generally, the values of C_p were somewhat lower than when m-phenylene diamine was used as a curing agent. In the third system (Figure 1) tris(dimethylaminomethyl)phenol tri(2-ethyl hexoate) was the curing agent and this system differed from the proceeding two in several respects. First, C_p was higher by about 10-15% over the temperature range of 40°C - 150°C, T_g occurred rather sharply and was complete at 70°C and C_p progressively increased to about 0.56 cal/gm°C at 150°C.

24. On the basis of these results, it would appear that the system Epox 828-tris(dimethylaminomethyl)phenol tri(2-ethyl hexoate) was less crosslinked than the other two systems on the assumption that C_p values are an index of the degree of crosslinking. The fact that T_g occurred sharply at a relatively low temperature reinforces this conclusion. Also, these results indicate the extent to which the physical properties of a resin system are dependent upon the curing agent used. In each case the stoichiometric amount of curing agent was used.

25. The systematic error in a typical determination of C_p as calculated in Equation 1 is estimated to be as follows:

<u>Quantity</u>	<u>Error</u>
I^2	1.5%
R	0.4%
M	0.2%
t	0.2%
T	<u>0.5%</u>
C_p	2.8%

From these estimated errors and from an analysis of the scatter of points on the C_p vs. temperature plots, the overall error is estimated to be within 5%. In addition, the accuracy of these C_p determinations has been checked by repeating the work of Wunderlich and Dole (Reference 3) on low pressure polyethylene. These investigators used an elaborate calorimeter capable of high accuracy and precision. The results of both sets of data are shown in Figure 6 and show excellent agreement. The values of C_p for polystyrene also show excellent agreement with the work of Ueberreiter and Otto-Laupenmuhlen (Reference 4).

CONCLUSIONS

26. The Cp results have led to the following conclusions:

- a. The Cp of thermosetting polymers is in the range 0.20 cal/gm°C to 0.60 cal/gm°C in the temperature range 30°C to 160°C and is temperature dependent.
- b. The Cp values obtained as a function of temperature by the adiabatic calorimeter are sensitive to second order transitions occurring within the polymer.
- c. The second order transition in thermosetting polymers generally occurs at a higher temperature than in thermoplastic polymers.
- d. A correlation exists between the second order transition and the heat distortion pc.nt.
- e. The Cp values are real functions of the structure of the polymer.

RECOMMENDATIONS

27. It is recommended that the Cp of polymers of known chemical and physical structure be determined. With data of this type it may be possible to relate the Cp values to the structure of the polymer. On the same polymers, the effects of varying amounts of curing agent and plasticizers on Tg should be established. Such information could be used to establish the nature of Tg. Finally, the change in Cp which occurs upon polymerization should be studied. This could be determined by measuring the Cp of both the monomer and polymer.

APPENDIX I

Polymers Studied

<u>Polymer</u>	<u>Type</u>	<u>Manufacturer</u>	<u>Designation on Figure</u>	<u>Curing Agent</u>
1. Epon 828	Epoxide	Shell Chemical Co.	Epon 828 + A	5.65% of diethylamino-propylamine
2. Epon 828	Epoxide	Shell Chemical Co.	Epon 828 + MPDA	12.6% of m-phenylene diamine
3. Epon 828	Epoxide	Shell Chemical Co.	Epon 828 + DMSA	56.5% of dodecyl succinic anhydride
4. Epon 828	Epoxide	Shell Chemical Co.	Epon 828 + D	11.5% of tris(dimethylaminomethyl)phenol-tri(2-ethyl hexoate)
5. Dow 2633	Epoxide	Dow Chemical Co.	Dow 2633 + D	10% of tris(dimethylamino-methyl)phenol tri(2-ethyl hexoate)
6. Epocast R-155	Epoxide	Furnace Plastics Co.	Epocast R-155	15% of a proprietary amine
7. Marlex 50	Polyethylene	Phillips Petroleum Co.	Polyethylene	-
8. Versamid 125(40%) ERL-2795(60%)	Polyamide-Epoxide Co-polymer	(General Mills Co.) Bakelite Corp.)	Polyamide-Epoxide	-
9. duPont 820	Filled Epoxide	duPont Co.	duPont 820-001	4.5% of piperidine
10. Hysol 6020-105	Filled Epoxide	Houghton Laboratories	Hysol 6020-105	11% of a diethylene triamine-epoxide adduct
11. Paraphex P-43	Styrene base Polyester	Rohm and Haas Co.	Paraphex P-43	2% of Cobalt naphthenate 1% of methyl ethyl ketone peroxide
12. Diallyl phthalate	-	Shell Chemical Co.	Diallyl phthalate	1.96% of benzoyl peroxide
13. Styron 666	Styrene	Dow Chemical Co.	Polystyrene	-

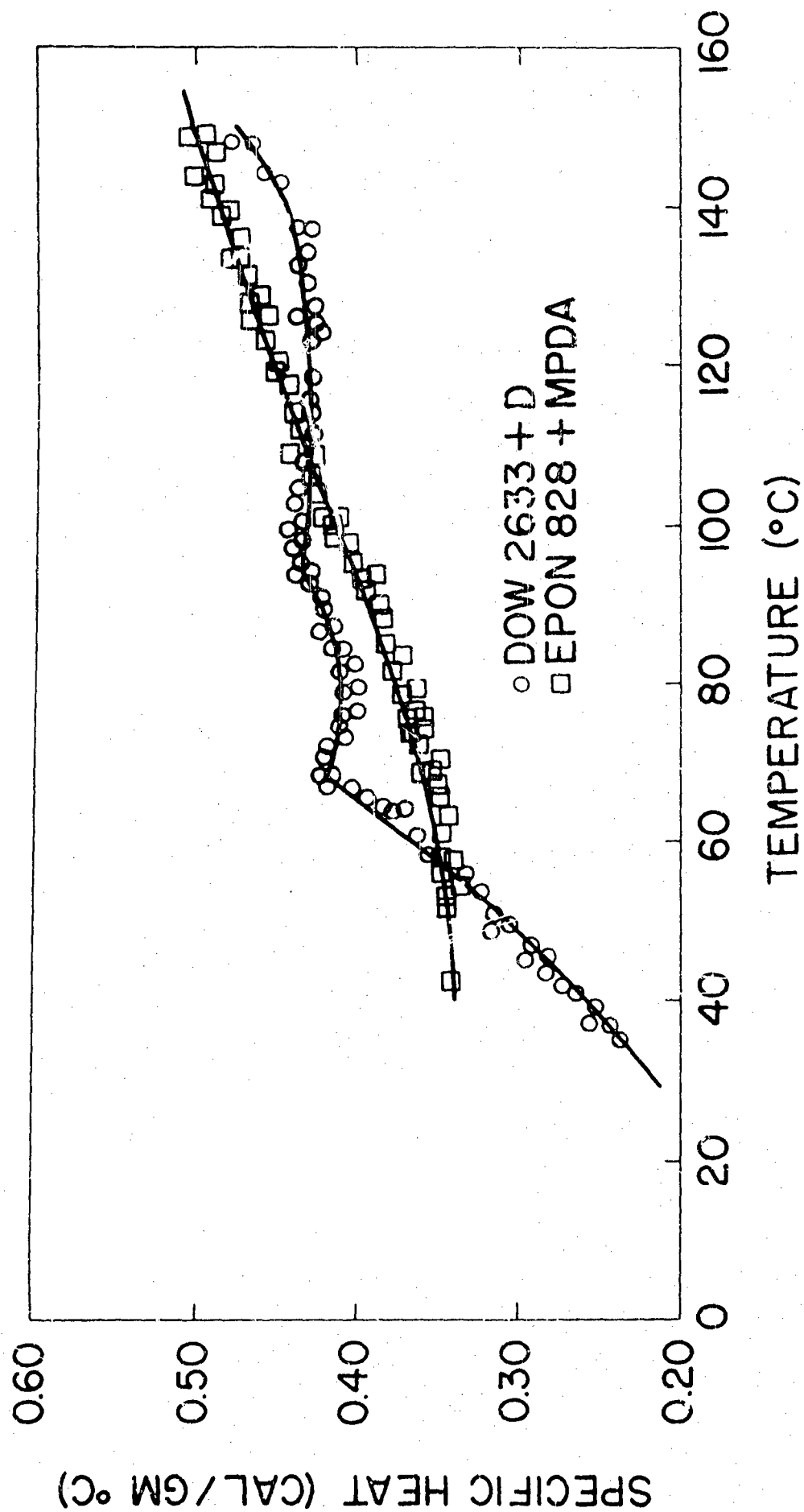


FIG. 1 SPECIFIC HEATS OF EPOXIDE POLYMERS

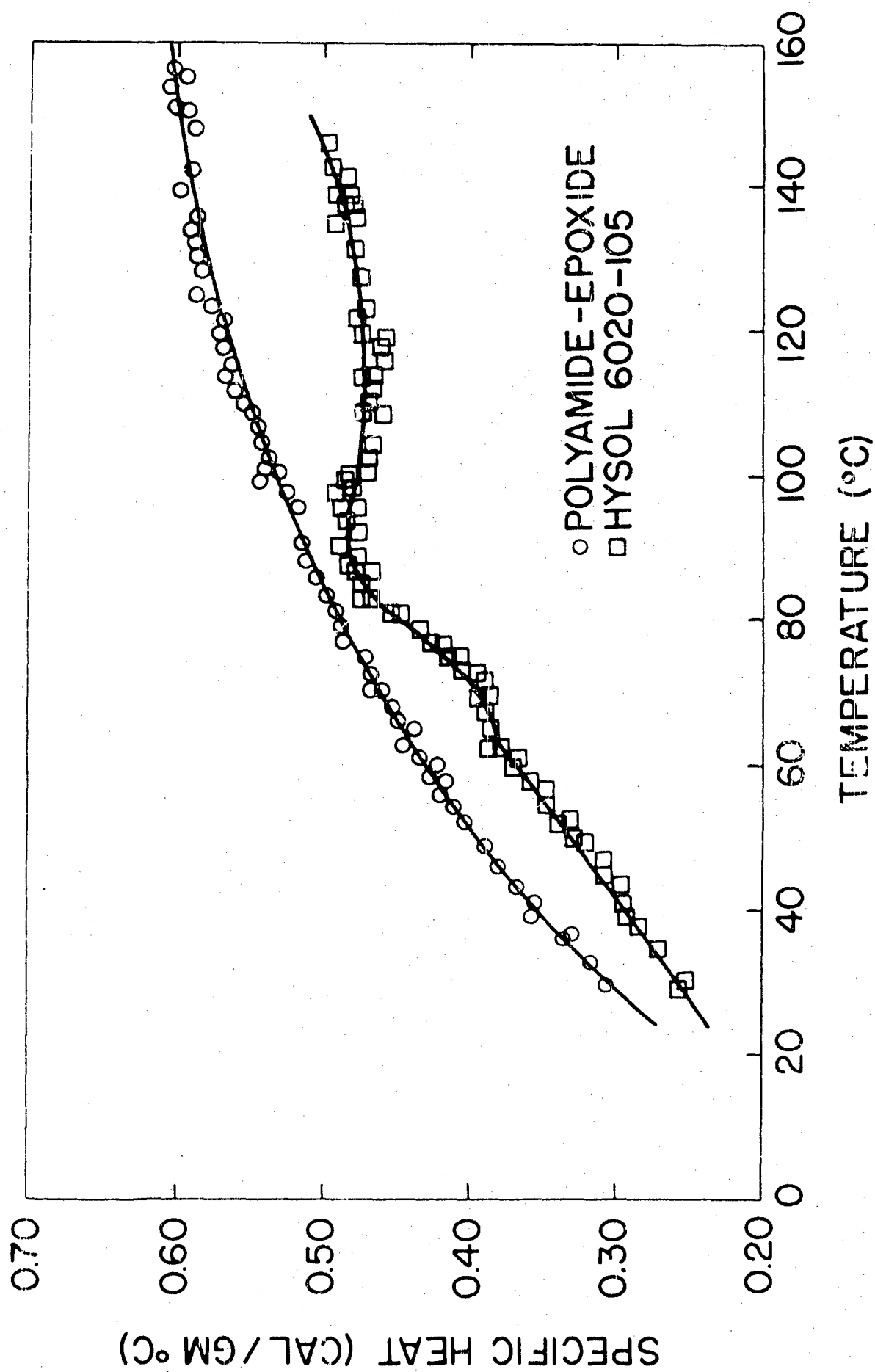


FIG. 2 SPECIFIC HEATS OF POLYAMIDE-EPOXYDE AND EPOXYDE POLYMERS

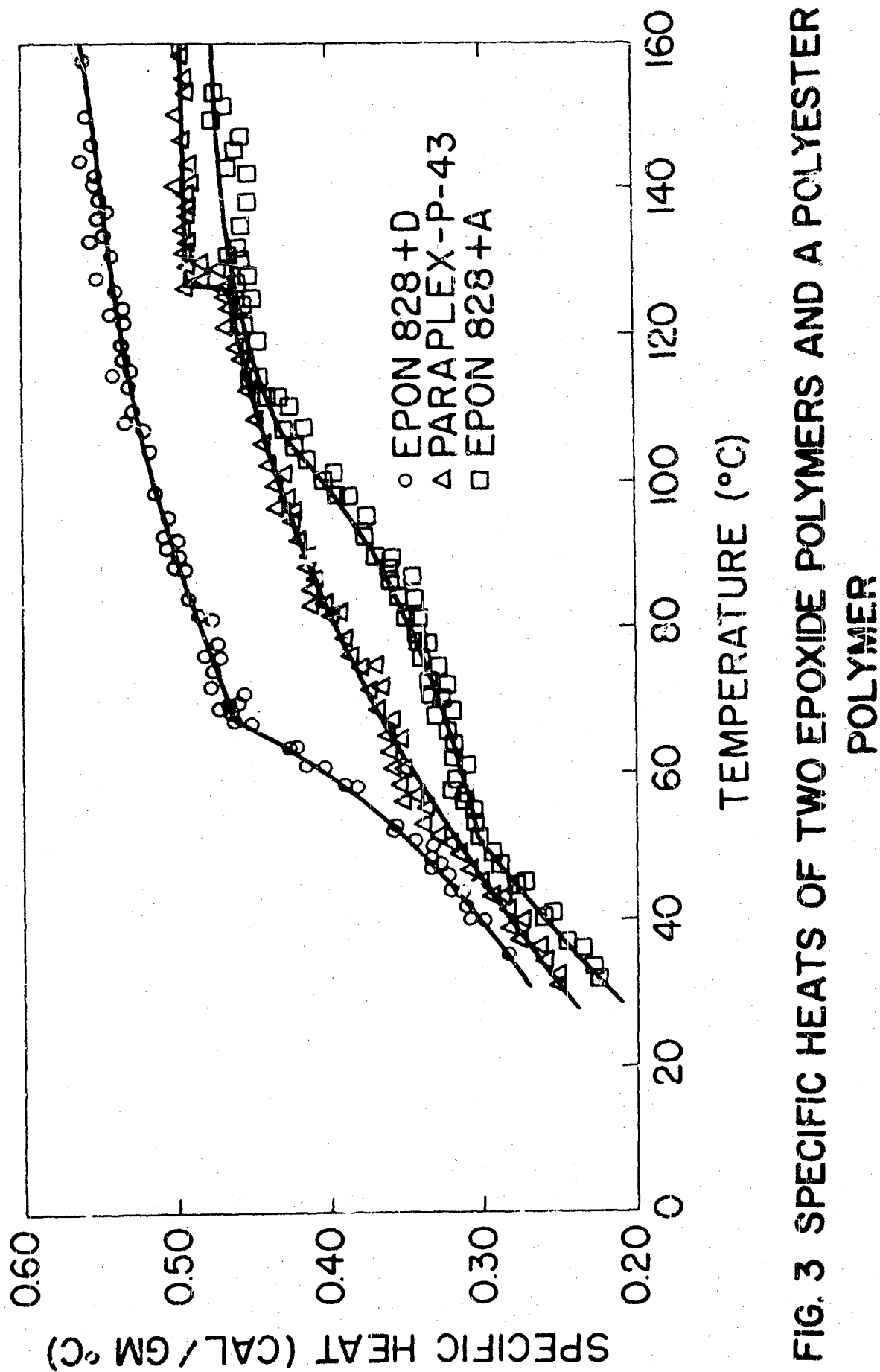


FIG. 3 SPECIFIC HEATS OF TWO EPOXIDE POLYMERS AND A POLYESTER

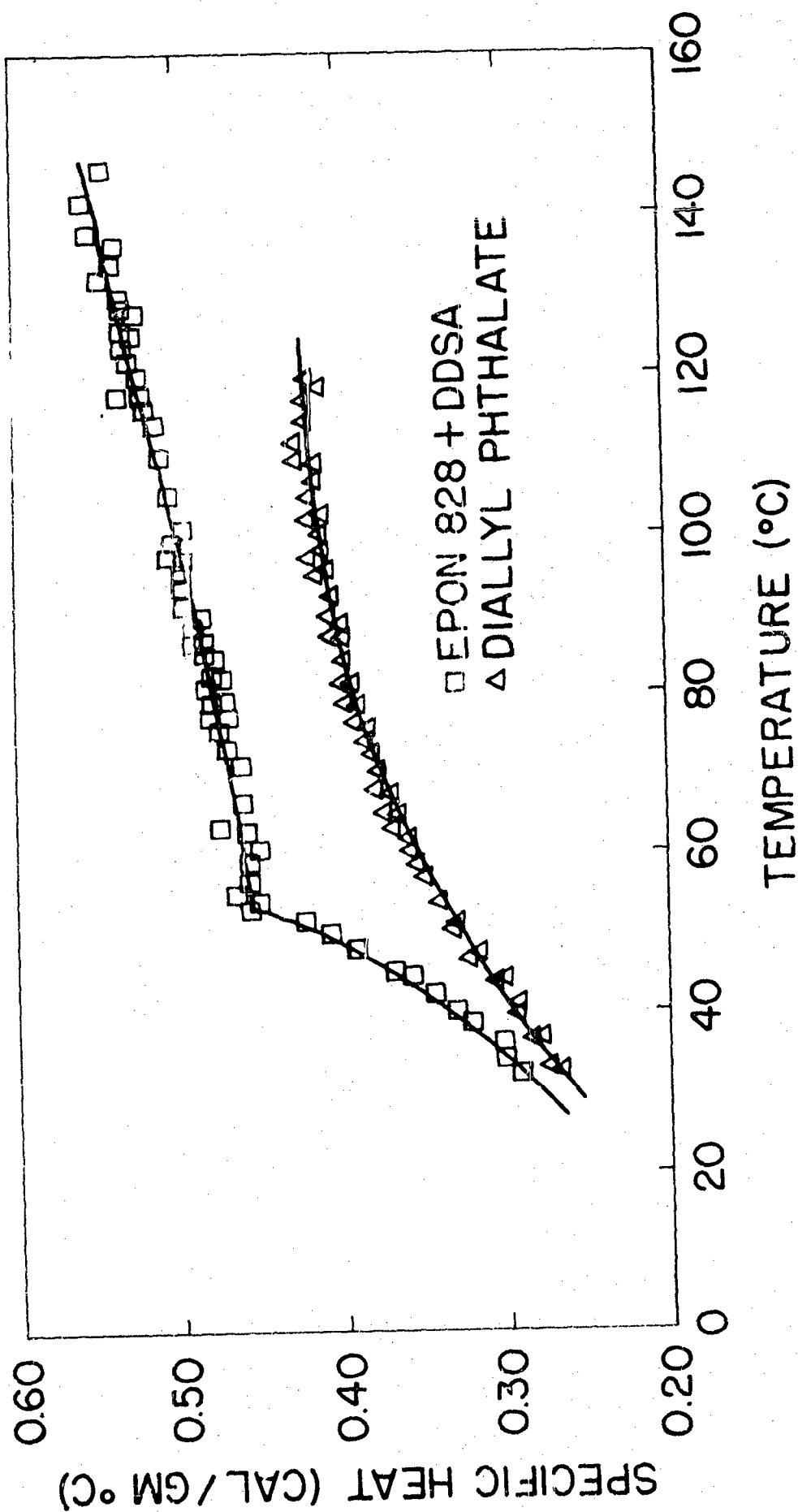


FIG. 4 SPECIFIC HEATS OF AN EPOXIDE POLYMER AND DIALLYL PHTHALATE

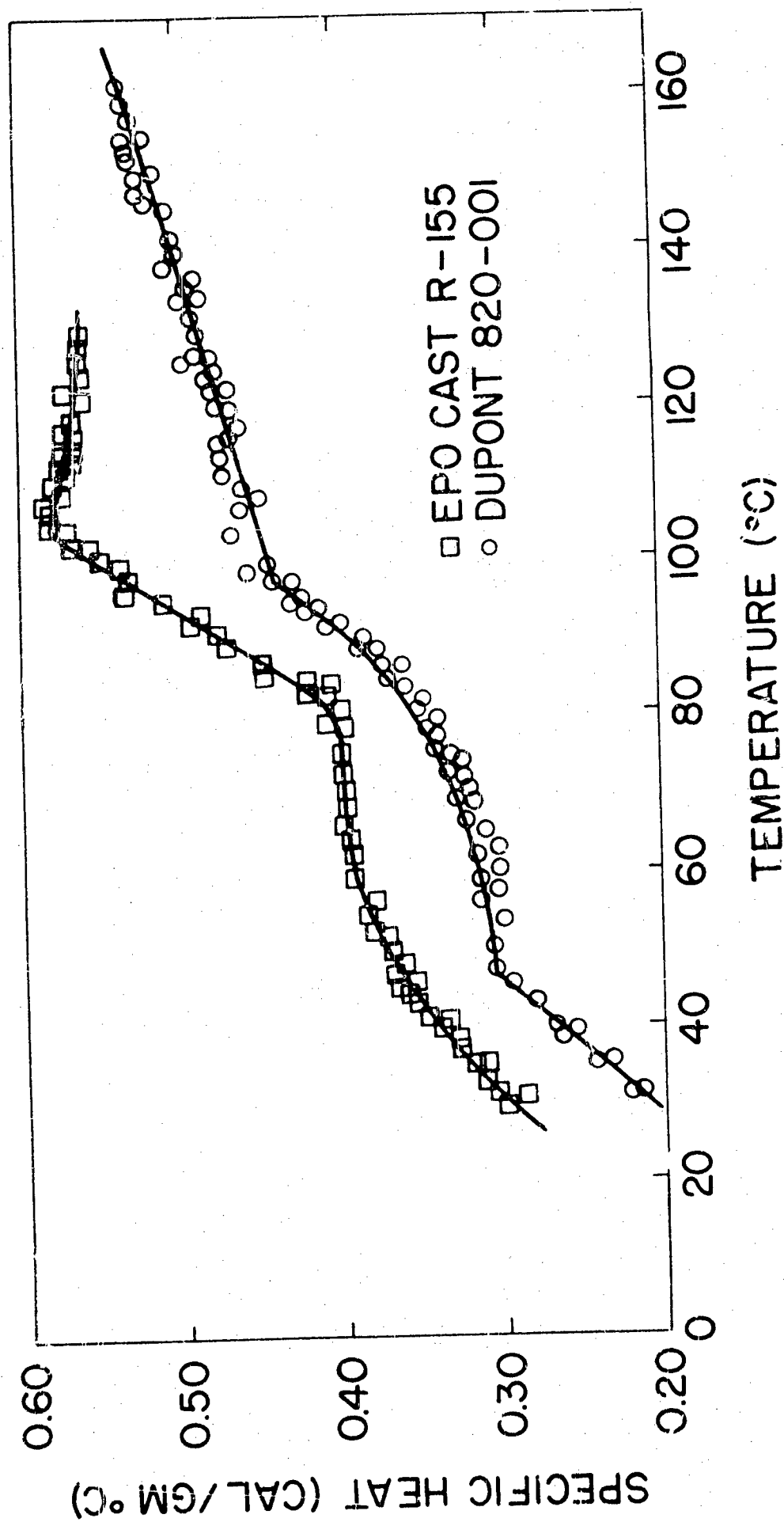


FIG. 5 SPECIFIC HEATS OF TWO EPOXIDE POLYMERS

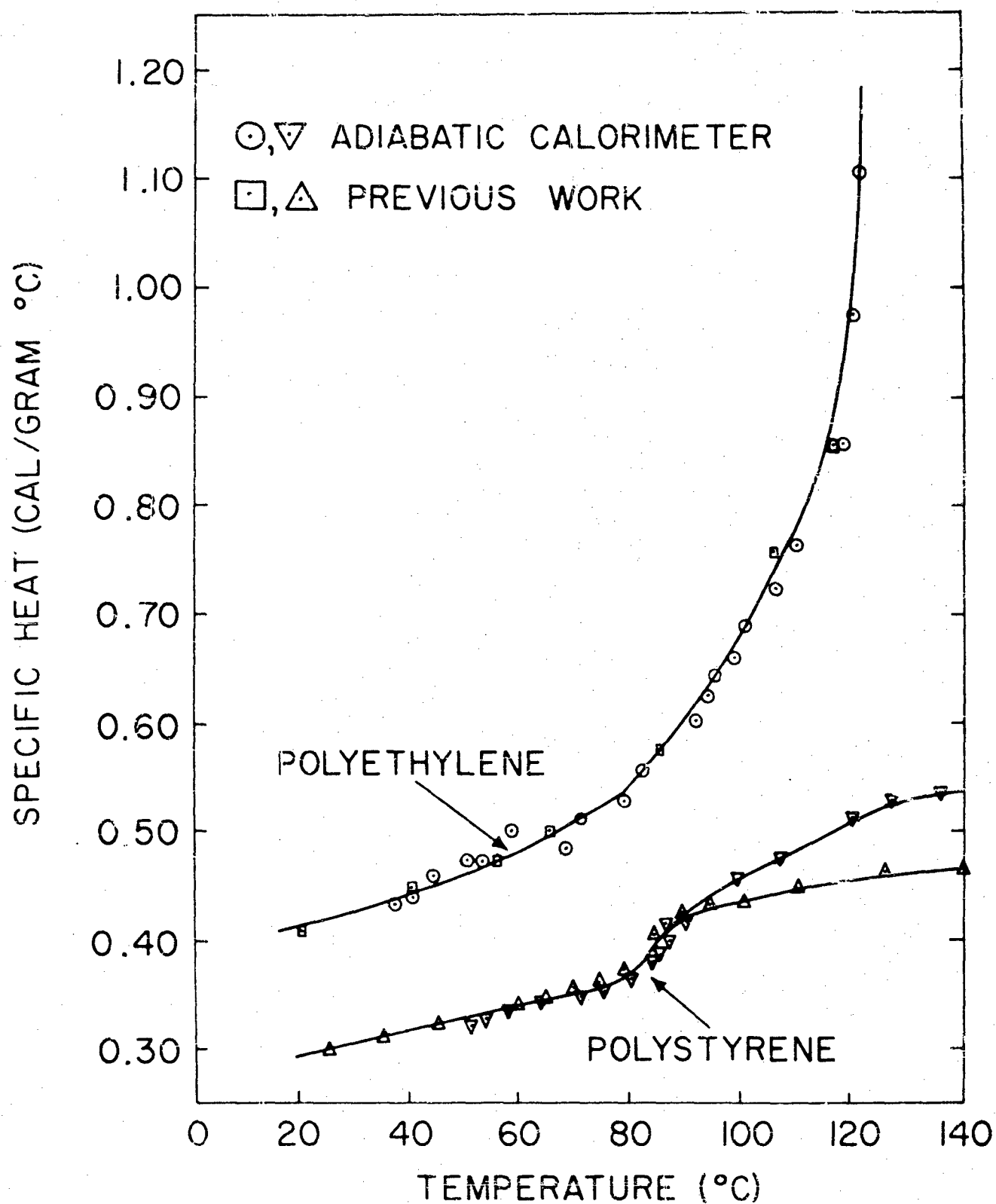


FIG. 6 SPECIFIC HEATS OF POLYSTYRENE AND POLYETHYLENE

TABLE I

TEMPERATURE RANGE OF SECOND ORDER TRANSITIONS, T_g

<u>Material</u>	<u>Figure Number</u>	<u>T_g Range</u>
Polyethylene	6	Not present
Epon 828 + DEAPA (1)	3	60°-115°C
Epon 828 + MFDA (2)	1	Not present
Epon 828 + DDSA (3)	4	40°-60°C
Epon 828 + DMP-30 (4)	3	40°-65°C
Dow 2633 + DMP-30 (4)	1	63°-80°C
Polyamide-ERL 2795	2	Not present
DuPont 820-001	5	45°-95°C
Epocast R-155	5	81°-110°
Hysol 6020-105	2	53°-85°C
Paraplex P-43	3	125°-128°C
Diallyl phthalate	4	Not present
Polystyrene	6	81°-82°C

- (1) Diethylaminopropylamine
 (2) m-phenylene diamine
 (3) Dodecenyl succinic anhydride
 (4) Tris(dimethylaminomethyl)phenol tri(2-ethyl hexoate)

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